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EMBRITTLMENT OF 4340 TYPE STEEL BY LIQUID LEAD AND
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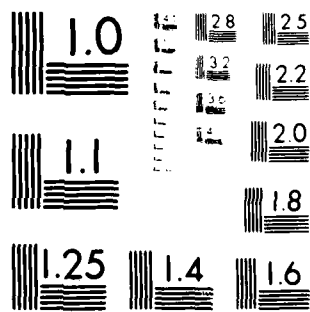
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TECHNICAL REPORT ARLCB-TR-84017

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**EMBRITTLEMENT OF 4340 TYPE STEEL
BY LIQUID LEAD AND ANTIMONY
AND LEAD-ANTIMONY SOLUTIONS**

M. H. KAMDAR

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**US ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER
LARGE CALIBER WEAPON SYSTEMS LABORATORY
BENET WEAPONS LABORATORY
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A study has been made of the fracture behavior of single-edge notched specimens of 4340 type steel tested in cyclic fatigue at temperatures ranging from 675°F to 1350°F in high purity lead and antimony and liquid lead containing 5 to 75 percent antimony in solution. The susceptibility to embrittlement by liquid lead decreases with temperature. The fracture mode changes from intergranular at low temperatures to ductile at 1200°F. In lead solutions, at low (CONT'D ON REVERSE)		

20. ABSTRACT (CONT'D)

temperatures (700°F) and low concentrations of antimony (5 to 25 percent), embrittlement is caused by liquid lead only. At high temperatures (1000 to 1200°F) and high concentrations of antimony (35 to 75 percent Sb), antimony is the primary embrittling species. Antimony induced embrittlement by lead-antimony solutions occurs by intergranular fracture mode. This variation in susceptibility to embrittlement with temperature indicates that at low temperatures embrittlement is caused by liquid lead and occurs by "reduction in cohesion" mechanism; while at elevated temperatures embrittlement is induced by antimony and occurs by temperature dependent grain boundary diffusion controlled processes. These and other results are also discussed in terms of the current understanding of liquid metal and temper embrittlement of metals.

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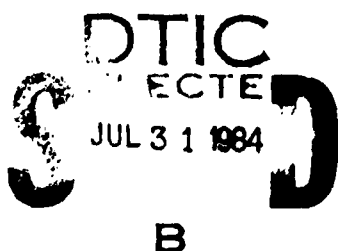
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INTRODUCTION

In many liquid-solid metal embrittlement couples, minor additions of certain elements to the embrittling liquid have been shown to change significantly the embrittlement susceptibility of the solid (ref 1). Such reports are, however, more intriguing than reliable because the investigations were not made with well characterized materials and under controlled conditions. It is not clear whether these effects attributed to additions of elements to the liquid may not be due to metallurgical, mechanical, or other factors. Recently, Breyer et al (ref 2) have made a systematic investigation of the effects of additions of antimony and tin to lead on the liquid lead embrittlement of steels as a part of their extensive investigations of embrittlement of internally leaded steels (ref 3) and also of steels embrittled by high purity liquid lead (ref 2).

It was shown that for smooth steel specimens tested in tension, the brittle fracture stress and the increase in the change in the brittle to ductile transition temperature increased linearly with antimony content of high purity liquid lead, Figures 1 and 2. However, tension tests with smooth specimens may not represent the intrinsic or the ultimate embrittlement susceptibility of steel in liquid lead or in lead containing minor additions of antimony. For instance, notched fatigue precracked steel specimens tested

¹W. Rostoker, J. M. McCaughey, and M. Markus, Embrittlement by Liquid Metals, Reinhold, New York, 1960.

²N. N. Breyer and K. L. Johnson, "Liquid Metal Embrittlement of 4145 Steel by Liquid-Tin and Lead-Antimony Alloys," Journal of Testing and Evaluation, Vol. 2, No. 6, American Society for Testing Materials, Philadelphia, 1974.

³S. Mostovoy and N. N. Breyer, "The Effects of Lead on the Mechanical Properties of 4145 Steel," Trans. Quart., 61, 1968, p. 219.

in liquid lead in cyclic fatigue failed at a stress intensity factor at failure which was one and one-half orders of magnitude lower in liquid lead than that in inert argon, whereas the fracture stress of smooth specimens in liquid lead was reduced by only forty percent from that in argon (ref 4). In another instance, Hayden et al (ref 5) showed that smooth specimens of high purity iron containing two to eight percent nickel were not embrittled by liquid mercury, whereas notched specimens of all composition (two to eight percent nickel) were severely embrittled by liquid mercury.

These investigations indicate that a fatigue test with a notched precracked or as notched specimen, rather than a tensile test with smooth specimen, provide a better measure of the intrinsic embrittlement susceptibility of a solid metal by a liquid metal. Therefore, fatigue tests with notched specimen should provide a better understanding of the true effects of additions of elements to an embrittling liquid on the change in the embrittlement susceptibility. Accordingly, in this investigation, we have used notched precracked specimens and subsequently, for the rest of the investigation, notched specimens tested in cyclic fatigue to investigate the effects of antimony additions to lead on the liquid lead embrittlement of steel. Full range of lead-antimony solutions and test temperatures were used so that the results can determine the effects of composition of liquid solutions on the variations in the brittle-ductile transition temperatures and

⁴M. H. Kamdar, "Embrittlement of Steel by Liquid Lead," Proceedings of Conference on Environmental Degradation of Engineering Materials, Virginia Polytechnic Institute, Blacksburg, VA, 1977, p. 235.

⁵H. W. Hayden and S. Floreen, Phil. Mag., Vol. 20, 1969, p. 235.

the fracture mode. These results are used to understand the role or effects of lead and antimony separately on the mechanism of embrittlement. Antimony is known to temper embrittle steel (ref 6), but direct evidence of antimony induced embrittlement is not available in the literature. Antimony induced temper embrittlement could be considered as solid antimony induced embrittlement of steel. This suggests that liquid antimony may also embrittle steel, since many metals which cause embrittlement in solid state also cause embrittlement in liquid state. Accordingly, steel specimens were tested in liquid antimony to provide direct evidence for antimony induced embrittlement.

EXPERIMENTAL

The material used in this investigation was high strength 4340 type steel and high purity (99.9999 percent) lead and antimony. The yield stress of steel at 675°F, was 125 Ksi and the fracture toughness at 675°F was 130 Ksi $\sqrt{\text{in}}$. From this steel, single edge notched fracture toughness specimens having ASTM configurations were machined for crack propagation studies in cyclic fatigue. The test specimens were 0.25 inches thick, 1.25 inches wide, and had loading pin holes four inches apart. They had a 60 degree notch with 0.005 inch root radius extending 0.525 inches through the width of the specimen. Some of the specimens were fatigued at 25°C using the procedure described elsewhere (ref 4) to introduce a sharp (about 0.04 inch long)

⁴M. H. Kamdar, "Embrittlement of Steel by Liquid Lead," Proceedings of Conference on Environmental Degradation of Engineering Materials, Virginia Polytechnic Institute, Blacksburg, VA, 1977, p. 235.

⁶B. J. Schulz and C. J. McMahon, "Alloy Effects in Temper Embrittlement," in Temper Embrittlement of Alloy Steels, STP 499, American Society for Testing Materials, Philadelphia, 1972, p. 104.

precrack. The same procedure was used to introduce fatigue precrack in specimens which were electroplated with a thin lead coating around the notch. The fatigue precrack was introduced underneath the lead plating thereby preventing oxidation or contamination of the precrack. This procedure assures wetting of the crack tip when lead is molten or liquid. Besides a few specimens containing a fatigue precrack, all other specimens used in this study had as-machined notch and did not have a fatigue crack at the root of the notch.

The specimens were cleaned in acetone, reverse etched, and using the procedure described previously (ref 4), were electroplated with 3 to 5 mil thick coating of lead extending one inch on either side and all around the notch in the specimen. The specimen was mounted in a stainless steel cylindrical environment test chamber. The environmental test chamber, 3 inches in diameter and 7 inches long, had a loading rod welded to its bottom. The loading rod extended inside the chamber. It had a slot at its end with holes where the specimen can be loaded by inserting a pin through the holes in the specimen and the loading rod. The rod extended outside the chamber and was threaded so that it can be screwed into the Sontag Fatigue testing machine. The cylinder was open at the other end and closed with a three inch diameter screw cap. The cap had an opening for the specimen so that it could be connected to the upper loading rod by inserting a loading pin through the specimen and the loading rod. The screw cap had an inlet coupling for

⁴M. H. Kamdar, "Embrittlement of Steel by Liquid Lead," Proceedings of Conference on Environmental Degradation of Engineering Materials, Virginia Polytechnic Institute, Blacksburg, VA, 1977, p. 235.

connecting argon gas line to provide continuous flow of argon inside the test chamber. The lead plated specimen was mounted in the environment chamber. A high purity lead, antimony, or lead-antimony solution of various compositions ranging from 5 to 75 wt. % antimony was melted in another stainless steel container and was poured around the specimen so that the liquid metal covered the entire specimen including the volume at least two inches above the notch of the specimen. The test chamber containing the specimen and solidified metal environment was screwed into the Sontag Fatigue testing machine. The upper end of the specimen was connected to the upper loading rod by inserting a pin through the holes in the specimen and the slot in the loading rod.

The cap was screwed on to the cylinder and the whole assembly was enclosed in a fast heating three zone split electric furnace. The heating of the three zones were controlled individually so that the liquid at the crack tip could be heated to 1200°F in one-half hour. The temperature of the metal bath was monitored by a thermocouple placed near the crack tip when the molten metal was poured earlier around the specimen. A small preload was applied to the specimen and was adjusted periodically so that the specimen would not break due to tensile stresses caused by the expansion of the specimen during heating or give erroneous results. When the liquid metal at the crack tip reached the desired temperature, the specimen was loaded to 1950 pounds for a tension-tension cyclic fatigue test. The specimen was tested at 1800 rpm in the Sontag machine to failure and the cycles to failure was recorded. The upper half of the specimen was lifted out of the molten bath immediately after failure by raising the upper loading rod to prevent excessive liquid metal from solidifying on the fracture surface. This facilitated subsequent removal

of the solidified liquid metal from the fracture surface. The fracture surfaces were cleaned by various means including chemical removal and film stripping of the adherent metal. The cleaned surface of an area was examined in a scanning electron microscope and the fracture mode was determined.

RESULTS

In the first series of tests, specimens containing fatigue precrack and as-notched specimens with lead plating failed in the same number of cycles as the specimens with no fatigue crack at the root of the notch. These results confirm earlier results on lead embrittlement of steel that failure in cyclic fatigue is independent of the root radii of a sharp fatigue crack produced at 20°C (refs 4,7). Therefore, all subsequent tests reported here used notched specimens which did not have fatigue precrack at the root of the notch. Each test data represents an average of three tests. The scatter in the results was small. The tension-tension cyclic fatigue test data for single-edge notched specimens tested in high purity (99.9999 percent) liquid lead, liquid antimony, and lead-antimony solutions containing 5, 25, 35, 50, and 75 wt. % antimony at temperatures ranging from 675°F to 1375°F are given in Table I.

The results in Table I show that embrittlement susceptibility of 4340 type steel by liquid lead decreases as the test temperature increases from 675°F to 1200°F. The failure mode changes from brittle intergranular, to

⁴M. H. Kamdar, "Embrittlement of Steel by Liquid Lead," Proceedings of Conference on Environmental Degradation of Engineering Materials, Virginia Polytechnic Institute, Blacksburg, VA, 1977, p. 235.

⁷M. H. Kamdar, "Embrittlement of Gun Steel by Liquid Lead," ARRADCOM Report ARLCB-TR-77046, Benet Weapons Laboratory, Watervliet, NY, December 1977.

ductile at 1200°F, Figures 3 and 4. Embrittlement by the ductile failure may occur in liquid metal embrittlement since the cycles to failure in liquid lead are some 50 percent lower than that in inert argon environment at the same temperature, i.e., 1200°F. Thus, lead may embrittle steel by brittle as well as ductile mode depending upon the test temperature. The ductile to brittle transition temperature in liquid lead is in the vicinity of 1200°F.

TABLE I. CYCLIC FATIGUE TEST DATA FOR AS-NOTCHED SPECIMENS TESTED AT 1950 LB. TENSION-TENSION LOAD IN ARGON, LIQUID LEAD, LIQUID ANTIMONY, AND LEAD-ANTIMONY SOLUTIONS OF VARIOUS COMPOSITIONS AT TEMPERATURES FROM 675°F TO 1375°F

Environment	Temperature °F	Cycles to Failure	Fracture Mode
Argon	1200°	30000	Ductile
Lead	675°	2000	Brittle
	900°	3000	Brittle
	1000°	5000	Semi-Brittle
	1200°	20000	Ductile
Lead-5 wt.% Sb	675°	2000	Brittle
	1000°	5000	Semi-Brittle
	1200°	20000	Ductile
Lead-25 wt.% Sb	675°	2000	Brittle
	1200°	1000	Brittle
Lead-35 wt.% Sb	675°	2000	Brittle
	1000°	1000	Brittle
	1250°	1000	Brittle
Lead-50 wt.% Sb	750°	2000	Brittle
	1000°	1000	Brittle
	1200°	500	Brittle
Lead-75 wt.% Sb	600°	2000	Brittle
	800°	2000	Brittle
	1000°	600	Brittle
	1200°	250	Brittle
Antimony (Liquid)	1200°	400	Brittle
	1375°	150	Brittle

Liquid lead containing five percent antimony to lead gave results which were the same as that for high purity liquid lead described above. Additions of five percent antimony to liquid lead had no effect on the susceptibility or the brittle-ductile transition temperature of steel in liquid lead. Additions of 25 to 75 percent antimony to lead and testing at 675°F, caused no change in the embrittlement susceptibility of steel by liquid lead.* At 1000°F, lead-antimony solutions containing 25 to 75 percent antimony increased embrittlement of steel. The number of cycles to failure decreased to 20 percent of that in liquid lead and remained essentially unchanged with increase in antimony content. At 1200°F (m.p. of Sb is 1177°F), which is also the brittle-ductile transition temperature of steel in liquid lead, lead-antimony solutions significantly increased embrittlement of steel. The embrittlement increased with increase in the antimony content of the lead-antimony solutions. The cycles to failure in lead-antimony (25-75 percent Sb) solutions were one to two orders of magnitude lower than that in the liquid lead, Table I. Also, at 1200°F, susceptibility to embrittlement increases with increasing antimony content. The cycles to failure decreases from 2000 for liquid containing 25 percent antimony to 250 for that containing 75 percent antimony. In liquid antimony, embrittlement was most severe and occurred in less than 200 cycles or in some fifteen seconds after the test was started. At all

*It should be noted that lead dissolves 28 percent antimony at 675°F. Additions of 28 to 75 percent Sb antimony results in a mixture of liquid containing 28 percent antimony and solid antimony. The liquid content decreases from 100 percent for 28 percent antimony to 20 percent for 75 percent antimony additions. These tests were performed to observe effects of solid antimony suspended in liquid lead on the embrittlement of steel.

test temperatures, specimens tested in lead-antimony solutions failed by brittle intergranular mode, Figures 5 through 8. The brittle fracture mode became more apparent as temperature increased and also as the antimony content of solutions increased. These results clearly demonstrate that at 1200°F embrittlement by liquid lead ceases, and embrittlement by antimony solutions becomes most severe.

DISCUSSION

The embrittlement of notched specimens tested in cyclic fatigue in high purity lead decreases with increase in temperature and the brittle to ductile transition occurs in the vicinity of 1200°F which is 500°F higher than that reported by Breyer (ref 2) for smooth 4145 steel specimens tested in tension in liquid lead. This significant increase in the transition temperature is related to the effects of cyclic fatigue tests vs. tensile tests on the embrittlement susceptibility. In a previous investigation by the author (ref 7), smooth specimens of the same steel used in this investigation tested in tension in liquid lead, failed at 100 Ksi, a stress which is close to the yield stress of 125 Ksi at 675°F. This stress is some 60 percent lower than the fracture stress of 250 Ksi in inert argon atmosphere. Similar results are reported by Breyer (ref 2) when smooth specimens of 4145 steel tested in tension in liquid lead at 700°F failed at 180 Ksi when the yield stress was 120 Ksi and the fracture stress in air was 225 Ksi. These tensile test

²N. N. Breyer and K. L. Johnson, "Liquid Metal Embrittlement of 4145 Steel by Liquid-Tin and Lead-Antimony Alloys," Journal of Testing and Evaluation, Vol. 2, No. 6, American Society for Testing Materials, Philadelphia, 1974.

⁷M. H. Kamdar, "Embrittlement of Gun Steel by Liquid Lead," ARRADCOM Report ARLCB-TR-77046, Benet Weapons Laboratory, Watervliet, NY, December 1977.

results for a steel similar to that used in this investigation are in agreement with each other, except that in the present investigation fracture occurred at or near yield stress, whereas in 4145 steel, the fracture stress was 50 percent higher than the yield stress. However, the embrittlement susceptibility increased significantly when notched specimens were tested in cyclic fatigue instead of smooth specimens in a tensile test. The stress intensity factor at failure in liquid lead at 675°F decreased by more than one order of magnitude from 135 Ksi $\sqrt{\text{in}}$ in argon to 7 Ksi $\sqrt{\text{in}}$ in liquid lead (ref 4). Thus, normally ductile steel becomes significantly brittle and notch sensitive in liquid lead environment. This significant increase in the embrittlement susceptibility would indicate that the transition temperature would be higher in cyclic fatigue tests than that in a tensile test as has been observed in this investigation.

It is apparent that cyclic fatigue tests with notched specimens rather than smooth tensile specimens provide a better measure of the inherent embrittlement susceptibility of steel in liquid lead. Another example of notch-sensitive embrittlement is found in the hydrogen embrittlement of nickel (ref 8). Smooth nickel single crystals tested in hydrogen at 20°C were not embrittled and failed by chisel type ductile failure. The same crystal containing a notch tested in hydrogen failed by quasi-cleavage type brittle

⁴M. H. Kamdar, "Embrittlement of Steel by Liquid Lead," Proceedings of Conference on Environmental Degradation of Engineering Materials, Virginia Polytechnic Institute, Blacksburg, VA, 1977, p. 235.

⁸M. H. Kamdar, "Embrittlement of Nickel by Gaseous Hydrogen," International Congress on Hydrogen in Metals, Chatenay-Malabry, France, 6-10 June 1977, Pergamon. Also in NASA Technical Report, 1975, Ames Research Center, Moffat Field, CA.

failure, and in argon by chisel type ductile failure. Furthermore, the increased susceptibility to embrittlement in a notched specimen tested in cyclic fatigue than that in a smooth specimen tested in tension can be interpreted as follows. In a smooth specimen, failure is nucleation controlled. In addition, embrittlement must occur at or above the yield stress because yielding* is a prerequisite for the occurrence of liquid metal embrittlement (ref 9). Plastic yielding is necessary to cause slip or dislocation pile-ups at a barrier such as a grain boundary. These pile-ups cause high stress concentrations at the barrier. Relaxation of stress concentrations can occur either by fracture or by plastic deformation and this will determine whether embrittlement will or will not occur. The yield stress decreases with increase in temperature. As a consequence, at elevated temperatures, relaxation of stress concentrations by plastic deformation will be preferred to crack nucleation and brittle fracture. Thus, embrittlement may not occur. On the other hand, presence of crack or a notch in a specimen means that stress concentrations are present at the crack tip prior to testing and a lower applied stress than that required to nucleate a crack in a smooth specimen will be needed for crack propagation. Some local yielding will occur at the crack tip. However, the applied stress is lower and therefore plastic blunting of crack tip will be less at elevated temperature. Therefore, in

*In some instances, sub-yield failure can occur as in course grain zinc in liquid mercury. Here, the critical resolved shear stress of the single crystal or yielding in a few grains rather than macroscopic yielding of the specimen is the prerequisite for liquid metal embrittlement (ref 9).

⁹M. H. Kamdar, "Embrittlement by Liquid Metals," Progress in Materials Science, Vol. 18, No. 6, 1973, pp. 289-374.

specimens containing a pre-existing crack, relaxation of stress concentration by brittle fracture in liquid metal environment will be preferred to plastic relaxation of stress concentrations by brittle failure. Thus, the conditions for the occurrence of embrittlement are most favorable in a precracked specimen tested in cyclic fatigue test than in a smooth specimen. As a consequence, an increase in the transition temperature will occur for notched specimens tested in cyclic fatigue.

Embrittlement of steel in lead-antimony solutions increases with an increase in temperature and becomes most severe at 1200°F; increases with antimony content of solutions, and is catastrophic in liquid antimony at 1200°F. The brittle-ductile transition temperature of steel in liquid lead is 1200°F. Therefore, embrittlement at 1200°F will not be caused by lead, but must be caused by antimony.

Antimony induced susceptibility to embrittlement which increases with temperature and antimony content is probably caused by temperature dependent processes such as diffusion, dissolution, or reaction at the crack tip. The dissolution at the crack tip will blunt the crack tip which will be further blunted by the enhanced plastic deformation that will occur with increasing temperature. Such a process will be unlikely to embrittle steel. On the other hand, diffusion of antimony in the grain boundaries will increase with temperature and the antimony content. Antimony induced weakening of large number of grain boundaries will cause embrittlement. Thus, embrittlement by antimony could occur by weakening of grain boundaries by temperature-composition dependent grain boundary diffusion controlled processes. The embrittlement susceptibility of steel in liquid lead decreases with tempera-

ture. Lead has virtually no solubility in steel in both the liquid and the solid state at all temperatures. In fact, liquid lead and liquid iron form two immiscible liquids. These observations and other reasons discussed in previous work (ref 4,7) suggested that embrittlement by liquid lead occurs by the classic "reduction in cohesion of atomic bonds at the crack tip" type of mechanism.

The embrittlement in lead-antimony solutions occurs by the synergistic effects of two different competing mechanisms, one controlled by liquid lead and the other by antimony in solution. At low temperature (600 to 700°F) and low concentrations of antimony (5 to 25 percent Sb) embrittlement is due to lead only. At high temperatures (1000-1200°F) and high concentrations of antimony (35 to 75 percent Sb), antimony is the primary embrittling species, although lead may have some effect.

Breyer et al (ref 2) have shown that additions of 0.02 to 0.2 percent antimony to lead significantly increases embrittlement susceptibility of smooth 4145 steel specimens tested in tension and the change in the transition temperature varies linearly with composition as shown in Figures 1 and 2. However, Musek and Breyer (ref 10) reported that linear correlation between

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- ²N. N. Breyer and K. L. Johnson, "Liquid Metal Embrittlement of 4145 Steel by Liquid-Tin and Lead-Antimony Alloys," Journal of Testing and Evaluation, Vol. 2, No. 6, American Society for Testing Materials, Philadelphia, 1974.
- ⁴M. H. Kamdar, "Embrittlement of Steel by Liquid Lead," Proceedings of Conference on Environmental Degradation of Engineering Materials, Virginia Polytechnic Institute, Blacksburg, VA, 1977, p. 235.
- ⁷M. H. Kamdar, "Embrittlement of Gun Steel by Liquid Lead," ARRADCOM Report ARLCB-TR-77046, Benet Weapons Laboratory, Watervliet, NY, December 1977.
- ¹⁰M. W. Musek and N. N. Breyer, "Effects of Alloy Additions to Tin on Metal Induced Embrittlement of High Strength Steel," Embrittlement by Liquid and Solid Metals, (M. H. Kamdar, ed.), AIME, May 1984.

transition temperature and antimony content did not occur when smooth 4145 steel specimens were tested in liquid tin containing various amounts (up to five percent) of antimony. Nevertheless, they suggest that adsorption of several monolayers of antimony atoms on crack surfaces and the crack tip could well provide a basis for the increase in embrittlement with solute content. Adsorption will increase with increase in the concentration of the antimony, but will decrease with increase in temperature, therefore, one may not observe increased embrittlement with increase in temperature. Thus, adsorption induced embrittlement is a less likely possibility. Diffusion of antimony in the grain boundaries will increase with increase in the antimony content and with increase in the temperature. Antimony induced weakening of the grain boundaries will facilitate crack nucleation and increase the susceptibility to embrittlement. A diffusion controlled process or mechanism will be consistent with increase in susceptibility with increase in antimony content and temperature reported in this investigation, Table I, and by Breyer et al (ref 2), Figures 1 and 2, and by Musek and Breyer (ref 10) for steel embrittled by tin-antimony solutions. The rate of diffusion of antimony in liquid lead or in liquid tin should be related to the differences in the embrittlement behavior of 4145 steel in lead-antimony and tin-antimony solutions observed by Breyer (ref 2) and Musek (ref 10).

²N. N. Breyer and K. L. Johnson, "Liquid Metal Embrittlement of 4145 Steel by Liquid-Tin and Lead-Antimony Alloys," Journal of Testing and Evaluation, Vol. 2, No. 6, American Society for Testing Materials, Philadelphia, 1974.

¹⁰M. W. Musek and N. N. Breyer, "Effects of Alloy Additions to Tin on Metal Induced Embrittlement of High Strength Steel," Embrittlement by Liquid and Solid Metals, (M. H. Kamdar, ed.), AIME May 1984.

The temperature range of 700 to 1200°F in which antimony is effectively the embrittling element in lead-antimony solutions is the same in which antimony is reported to temper embrittle steel (refs 6,11). This suggests that a common mechanism may exist for both the liquid and temper embrittlement of steel by antimony. Embrittlement of steel by liquid antimony provides direct evidence that antimony is the embrittling element for steel. In many instances (ref 12), certain elements cause both the liquid and solid metal induced embrittlement of metals. Thus, temper embrittlement of steel could very well be considered as solid antimony induced embrittlement. The extent of embrittlement or the brittle to ductile transition in antimony may be related to the grain boundary diffusion characteristics of liquid and solid antimony in steel.

SUMMARY

1. The brittle-ductile transition temperature of steel specimen in liquid lead is 1200°F which is 500°F higher than that reported for smooth specimens tested in tension.
2. At low temperatures (600 to 700°F) and for concentrations of antimony of up to 28 percent in lead-antimony solutions, embrittlement is caused by lead only.

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- ⁶B. J. Schulz and C. J. McMahon, "Alloy Effects in Temper Embrittlement," in Temper Embrittlement of Alloy Steels, STP 499, American Society for Testing Materials, Philadelphia, 1972, p. 104.
- ¹¹J. Kameda and C. J. McMahon, Jr., "Solute Segregation and Brittle Fracture in Alloy Steel," Met. Trans. Vol. 11A, 1980, p. 91.
- ¹²A. Druschitz and P. Gordon, "Solid Metal Induced Embrittlement of Metals - A Review," Embrittlement by Liquid and Solid Metals, (M. H. Kamdar, ed.), AIME, May 1984.

3. Lead embrittlement of steel occurs by the "reduction in cohesion of atomic bonds at the crack tip" mechanism.

4. At high temperatures (1000 to 1200°F) and high concentrations of antimony (35 to 75 percent Sb), embrittlement in lead-antimony solutions occurs by the synergistic effects of both lead and antimony, however, embrittlement is primarily caused by antimony.

5. Liquid antimony embrittles steel.

6. Antimony induced embrittlement probably occurs by temperature and antimony concentration dependent processes of grain boundary diffusion of antimony causing weakening of the grain boundaries.

7. Antimony induced embrittlement occurs in the same temperature range in which antimony temper embrittles steel. This suggests that some common process may cause both liquid and temper embrittlement of steel, and that temper embrittlement may be considered solid antimony induced embrittlement of steel.

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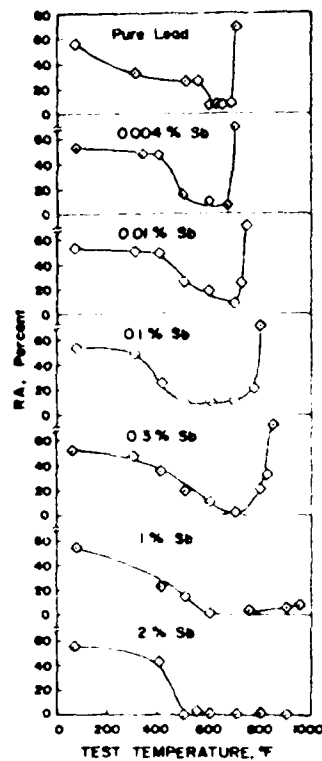


Figure 1. Reduction-in-area for 4145 steel surface wetted with lead and various lead-antimony alloys as a function of test temperature.

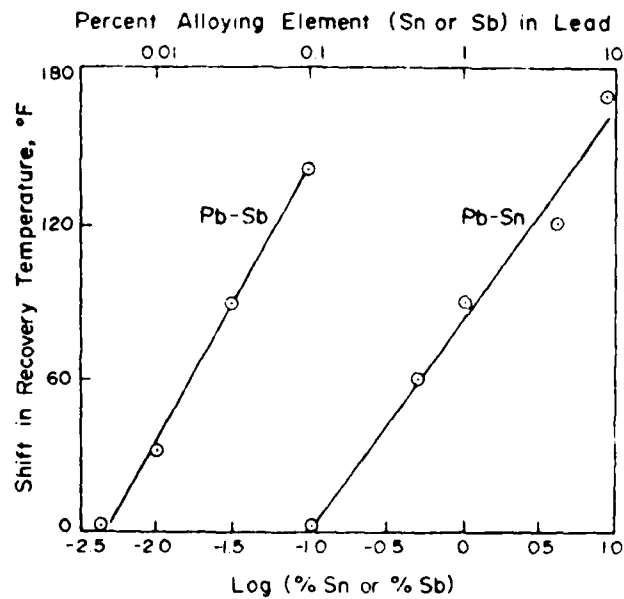


Figure 2. Relationship between shift in recovery temperature and antimony and tin in lead.

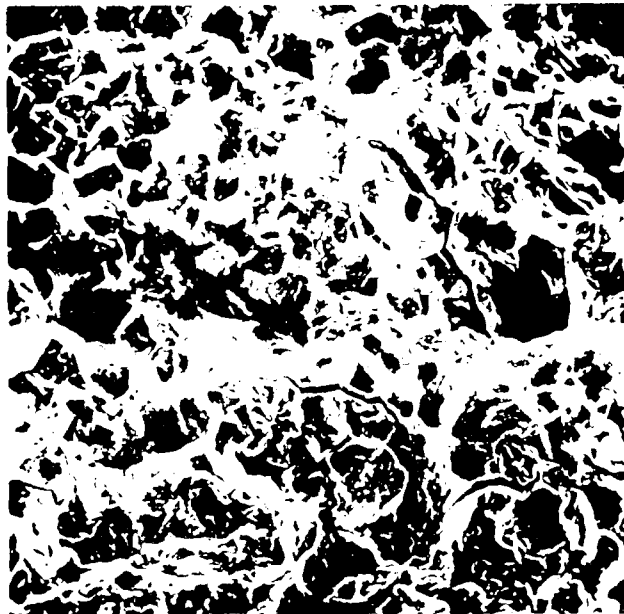


Figure 3. SEM fracture surface of a specimen after a 1000 psi liquid burst test in 1100F deaerated water.

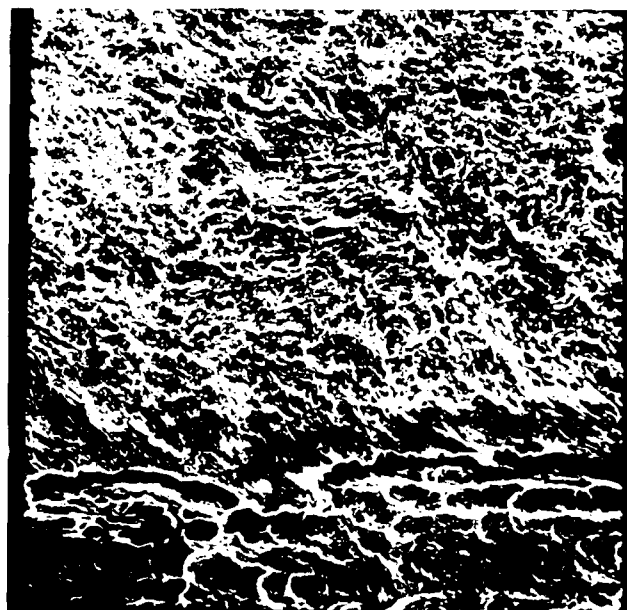


Figure 4. SEM fracture surface of a specimen after a 1000 psi liquid burst test in 1100F deaerated water.

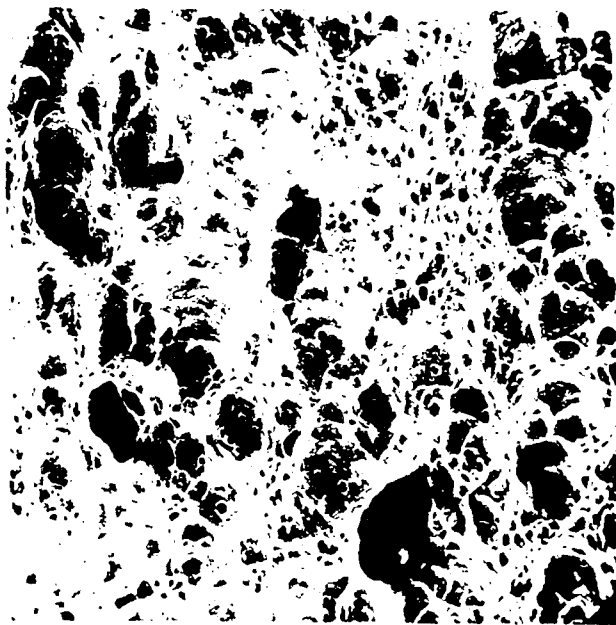


Figure 5. SEM fractograph showing surface features in a specimen tested in air at 1200°C, air, 100x.



Figure 6. SEM fractograph showing surface features in a specimen tested in 20% 2H₂-75% air at 1200°C, air, 100x.

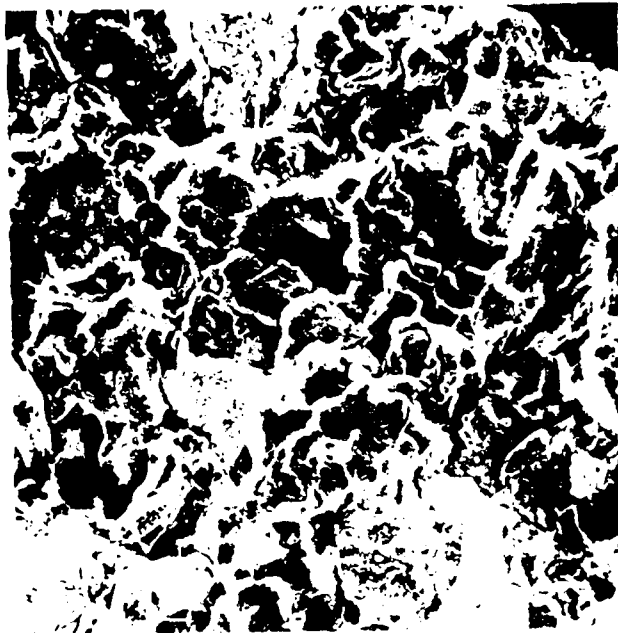


Figure 7. SEM fractograph showing intergranular cracks in a specimen tested in 50% Pb-50% Sb solution at 1000°F, Mag. 500X.

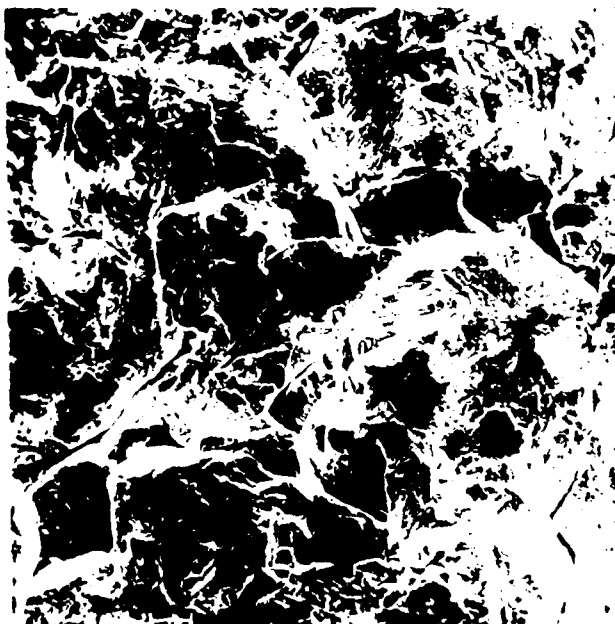


Figure 8. SEM fractograph showing brittle intergranular cracks in a specimen tested in liquid tin-bismuth at 1200°F, Mag. 500X.

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